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Impact of crude vegetable oils on the oxidation reactivity and nanostructure of diesel particulate matter

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ABSTRACT

This paper presents the chemical composition, oxidation reactivity and nanostructural characteristics of the particulate matter (PM) produced by a diesel engine operating with crude palm oil, crude jatropha oil and commercial diesel fuel. The engine was operated under a stationary mode of 25 N m and 1800 min⁻¹. Non-diluted PM was collected through a stainless steel filter located 1.5 m downstream from exhaust manifold. The soot (devolatilized PM) was subjected to TGA, FTIR, DRIFT and Raman spectroscopy, while the raw PM was subjected to TEM, SEM and XRD spectroscopy. A significant difference between diesel and crude vegetable oil (CVO) soot composition was observed. C=O bonds and C=C stretching mode were higher for CVOs compared with diesel soot. However, the contrary trend was observed with the C–H aromatic groups (3030 cm⁻¹), which were measured by DRIFT spectroscopy. Both CVOs PM samples showed a higher volatile organic fraction (VOF, almost the double), a lower fringe length (L_a) and a lower stacking thickness (L_c) than those of the diesel PM. Jatropha oil, the most reactive of all soot samples, presented the highest active surface area (ASA), the lowest L_a and L_c and the most ordered graphite-like nanostructure. Palm oil, which has the highest interplanar distance (d_{002}) and the smallest geometric primary particulate diameter, exhibited the highest concentration of aliphatics by mass. The graphite-like structure of both CVOs soot was more ordered than of the diesel soot. This result is in agreement with the increase in the C=C stretching mode of both CVOs soot observed by FT-IR. Fractal dimension of diesel was higher than that of both CVOs soot samples. Under this test condition and engine configuration, it can be concluded that the use of CVOs affects the chemical composition of PM and the oxidation reactivity and nanostructure of the soot.

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1. Introduction

The crude vegetable oil (CVO) could be an attractive alternative as partial or even total substitute for diesel fuel under some special conditions such as small-scale on-farm power generation, agricultural areas not connected to the national electrical grid, or in tractor's engines operating in oil plantations, where the diesel fuel is scarce and expensive. The small-scale CVO production for agricultural machinery and tractors has proven to be feasible, from the economic and technical point of view in certain Spanish areas [1] and in the United States of America [2]. Using the framework of Life Cycle Analysis (LCA), Esteban et al. [3] found that the energy return on investment (EROI) and energy conversion ratio indices were more favorable for small-scale CVO than for biodiesel, in terms of the energy used. It was explained because the production

of biodiesel requires more processes that are more complex and energy intensive than the CVO production.

Engine performance, combustion and gaseous emissions of small diesel power plants operating with CVO have been widely studied. Srivastava and Prasad [4] reviewed main technical concerns related to the effects of triglyceride-based diesel fuels properties on the engine performance, emissions and durability issues. They pointed out how injection, atomization and combustion characteristics could be affected by CVO properties, mainly its high viscosity, low volatility, low cetane number, and high tendency to oxidative and thermal polymerization. This leads to a series of long-term engine operation problems, which requires the CVO to be modified according to the conditions of use. Some researchers have successfully implemented the preheating and/or filtering of the CVO before entering the engine [5–7]. Blending strategy with diesel fuel has proven to be adequate for this purpose [8–11]. Some efforts in the definition of quality standards for CVO have been recently proposed [12]. For example, in Germany, the standard

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for quality requirements “Rapeseed fuel for Diesel engines”, has been recently published as an official DIN standard (DIN 51605:2010-10) [13].

The nanostructure and oxidation reactivity of diesel soot have been analyzed with different techniques such as those reported by Lapuerta et al. [14]. Although both parameters have been widely studied for other alternative fuels such as hydrotreated vegetable oil [15] and biodiesel fuels [14,16–25], these studies have not compared particulate matter (PM) from diesel and CVO fuels. This is because COV has not been commonly used in commercial diesel engines. Nevertheless, and as mentioned above, this alternative fuel has been gaining interest over the past years. The study of the chemical composition, nanostructure and oxidation reactivity of PM produced by CVOs may contribute to the knowledge of design parameters of diesel particulate filters, and to determine its potential as a human health hazard.

PM nanostructure is closely related to oxidation reactivity and they are governed by the fuel properties and combustion process under which the PM is formed. High temperature, long residence time and rich local fuel environment, promote pyrolysis kinetics leading to polyaromatic hydrocarbons (PAHs), which are the main constituents of the graphene layers. During PM formation, oxygenated functional groups, like C–OH and C=O, aliphatic C–H groups and several impurities, that affect soot reactivity, are attached to non-six membered PAHs rings. Although the relation between carbon structure and reactivity has been widely studied and is well known for carbon science, still remains unclear for diesel soot, especially for bio-based diesel fuels.

With regard to the effect of fuel on soot properties, Vander Wal and Tomasek [26] used a high temperature tube furnace to demonstrate that acetylene-derived soot had a more graphitic structure and was less reactive (by nearly fivefold) than benzene-derived soot. The curvature of carbon layers was found to substantially increase oxidation reactivity, as observed for ethanol-derived soot. They also found that the fuel controls the soot nanostructure under specific combinations of temperature and residence times [27]. Song et al. [19] reported that neat soybean oil biodiesel (NSB) soot, collected from a 5.9 L diesel engine, was more reactive than soot from Fischer–Tropsch (FT). At a first instance, they claimed that oxidation reactivity of the soot was more dependent on the relative amount of initial surface oxygen groups than on its initial structure properties. However, they reported later in a separate work, that those groups, as determined by XPS, did not dominate the soot oxidation reactivity [18,28] and that even under matched combustion phasing, fuel formulation such as oxygen and aromatics content, had a definitive influence on soot reactivity and nanostructure [18]. They also found that NSB exhibited faster oxidation than low sulfur diesel (BP15) and FT soot, while fringe length (L_n) followed an inverse relation to the oxidation reactivity [18]. By experimental and numerical approaches, Mueller et al. [29], showed that soot and soot-precursors formation characteristics might be different depending on the oxygenated fuel used in a single cylinder diesel engine. They found that tri-propylene glycol methyl ether (TPGME) was more effective at reducing soot than that containing di-butyl maleate (DBM). They also observed that fuel oxygenation was more effective at reducing soot than enhancing the entrainment of oxygen from charge gases.

The effect of combustion process on PM properties has been studied by several researchers. Lee et al. [30] collected PM from a 2.4 L single-cylinder supercharged direct injection diesel engine. They found that the engine load (in-cylinder pressure and temperature) was more important for the formation of particulates agglomerates than the engine speed (characteristic time). The primary particulate size decreased with the engine load and speed, and the particulates turned more ordered and partially oxidized due to the high in-cylinder temperature. Neer and Koylu [31]

observed, by using a 5.9 L, 6-cylinder medium-duty diesel engine, an increase in soot spherule and aggregate size with the engine load. The contradiction with the study conducted by Lee et al. [30] could be attributed to differences in engine technology and experimental conditions. Zhu et al. [32] observed that the degree of order of the soot structure increased with the engine load, and that soot particulate size decreased due to the faster oxidation of the particulate at high in-cylinder temperature. Al-Qurashi and Boehman [33] using a 2.5 L, 4-cylinder, turbocharged, common rail direct injection diesel engine, found that soot generated under 20% of EGR was more reactive due to slow external burning and rapid internal burning as compared to non-EGR soot, which burned from outside in.

Chien et al. [17] used FTIR spectroscopy to determine the composition of the PM produced by a non-road diesel generator operating with NBS. They found that chemical structures of PM were closely related to the fuel and engine oil properties, and that engine load had not a significant effect on the PM composition. Li et al. [34] used a 5.79 L, 6-cylinder, common rail, direct injection, heavy-duty diesel engine modified to develop a total cylinder sampling system (TCSS) to collect in-cylinder soot generated during combustion process. They found that both fringe tortuosity and separation distance decreased as combustion proceeds, indicating that the soot evolved towards a more graphitic structure during the combustion process. Wang et al. [35] used the same experimental set-up that the one used by Li et al. [34] and found that independently of the equivalence ratio, the relative amount of aliphatic C–H groups present in the diesel soot surface was more important in governing soot oxidation reactivity than C–OH and C=O groups. Lu et al. [36] using a naturally aspirated, 4-cylinder, direct injection diesel engine found that primary particle size was not affected by the engine operating conditions. They observed that graphitization of primary particles was affected by the engine torque, but not by the engine speed. In contrast, Yehliu et al. [37] showed that the impact of the engine speed (characteristic time) was more pronounced than the impact of the engine torque (equivalence ratio), with regards to soot oxidation reactivity and nanostructure.

This work examines the impact of the crude jatropha and palm oils on the chemical composition, oxidation reactivity and nanostructure of the PM produced by a small diesel power plant. PM properties were explained by means of experimental combustion diagnosis, based on instantaneous in-cylinder pressure and fuel properties. TGA was used to characterize soot oxidation reactivity and active surface area (ASA); and to determine PM composition by proximate analysis (VOF, fixed carbon and residues). FTIR and DRIFT spectroscopy were used to identify surface functional groups of soot samples. Raman spectroscopy was used to determine the graphite-like structure of the soot, and XRD spectroscopy was used to quantify interplanar distance between graphene layers and stacking thickness and length of fringes. Finally, TEM and SEM were used to determine the geometric diameter of the primary particles and the morphology of the agglomerates, respectively.

2. Methodology

2.1. Engine parameters and sampling procedure

All tests were carried out in an instrumented naturally aspirated, two-cylinder, air cooled, direct injection diesel engine typically used in agriculture or in small power plants. The engine was coupled to a hydraulic dynamometer brake (Go-Power D512). Table 1 lists the main characteristics of the engine. The temperature of the air-cooling was kept constant to 30 °C through two electrical resistances. The engine test bed was instrumented with

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